

# WEST Search History

DATE: Tuesday, June 18, 2002

## Set Name Query side by side

## Hit Count Set Name result set

*DB=USPT; PLUR=YES; OP=OR*

L37	estee.as.	68	L37
L36	(interference near5 pigment\$ ).ab. and (interference near5 pigment\$ near30 green) and cosmetic	9	L36
L35	L34 and cosmetic	16	L35
L34	(interference near5 pigment\$ ).ab. and green	32	L34

*DB=JPAB,EPAB,DWPI; PLUR=YES; OP=OR*

L33	L32 and cosmetic	2	L33
L32	(interference near5 pigment\$ ) and green	13	L32
L31	(green near100 platelet\$ near100 pigment\$) and cosmetic	1	L31

*DB=USPT; PLUR=YES; OP=OR*

L30	(green near100 platelet\$ near100 pigment\$) and cosmetic	6	L30
L29	green and platelet\$ and pigment\$ and cosmetic	221	L29
L28	green and platelet\$	5075	L28
L27	green.ab. and cosmetic and skin	35	L27
L26	green.ab. and (titanium adj oxide) and cosmetic and skin	0	L26
L25	green and (titanium adj oxide) and cosmetic and skin	238	L25
L24	green and (titanium adj oxide)	5954	L24
L23	(polymethylsilsesquioxane polymethylsilsesquixane) and (aging skin wrinkl\$ lines)	1	L23

*DB=JPAB,EPAB,DWPI; PLUR=YES; OP=OR*

L22	(polymethylsilsesquioxane polymethylsilsesquixane)	2	L22
L21	(polymethylsilsesquioxane polymethylsilsesquixane) and skin	0	L21
L20	(polymethylsilsesquioxane polymethylsilsesquixane) and wrinkle	0	L20

*DB=USPT; PLUR=YES; OP=OR*

L19	(polymethylsilsesquioxane polymethylsilsesquixane) and wrinkle	0	L19
L18	(polymethylsilsesquioxane polymethylsilsesquixane) and (refractive adj index)	0	L18
L17	polymethylsilsesquioxane.ab.	1	L17
L16	4892726.pn.	1	L16
L15	L14 and ((424/\$).ccls.)	0	L15
L14	polymethylsilsesquioxane.ab.	15	L14
L13	polymethylsilsesquioxane.ab. and cosmetic	3	L13
L12	L11 and cosmetic	17	L12
L11	polymethylsilsesquioxane	141	L11

L10	L8 and polymethylsilsesquioxane	0	L10
L9	L8 and green	4	L9
L8	shiseido.as. and (anatase )	7	L8
L7	shiseido.as. and (anatase adj titanium adj dioxide)	1	L7
L6	L5 and matt\$	16	L6
L5	cosmetic and (timiron flamenco)	57	L5
L4	shiseido.as. and (timiron flamenco)	1	L4
L3	L2 and reflect\$	11	L3
L2	L1 and (TIO\$ titanium )	46	L2
L1	shiseido.as. and green and pigmen\$	53	L1

END OF SEARCH HISTORY

# WEST Search History

DATE: Tuesday, June 18, 2002

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*DB=USPT; PLUR=YES; OP=OR*

L23	(polymethylsilsesquioxane polymethylsilsesquixane) and (aging skin wrinkl\$ lines)	1	L23
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*DB=JPAB,EPAB,DWPI; PLUR=YES; OP=OR*

L22	(polymethylsilsesquioxane polymethylsilsesquixane)	2	L22
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L21	(polymethylsilsesquioxane polymethylsilsesquixane) and skin	0	L21
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L20	(polymethylsilsesquioxane polymethylsilsesquixane) and wrinkle	0	L20
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*DB=USPT; PLUR=YES; OP=OR*

L19	(polymethylsilsesquioxane polymethylsilsesquixane) and wrinkle	0	L19
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L18	(polymethylsilsesquioxane polymethylsilsesquixane) and (refractive adj index)	0	L18
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L17	polymethylsilsesquioxane.ab.	1	L17
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L16	4892726.pn.	1	L16
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L15	L14 and ((424/\$).ccls.)	0	L15
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L14	polymethylsilsesquioxane.ab.	15	L14
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L13	polymethylsilsesquioxane.ab. and cosmetic	3	L13
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L12	L11 and cosmetic	17	L12
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L11	polymethylsilsesquioxane	141	L11
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L10	L8 and polymethylsilsesquioxane	0	L10
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L9	L8 and green	4	L9
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L8	shiseido.as. and (anatase )	7	L8
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L7	shiseido.as. and (anatase adj titanium adj dioxide)	1	L7
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L6	L5 and matt\$	16	L6
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L5	cosmetic and (timiron flamenco)	57	L5
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L4	shiseido.as. and (timiron flamenco)	1	L4
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L3	L2 and reflect\$	11	L3
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L2	L1 and (TIO\$ titanium )	46	L2
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L1	shiseido.as. and green and pigmen\$	53	L1
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END OF SEARCH HISTORY

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L45: Entry 4 of 5

File: USPT

May 28, 1996

DOCUMENT-IDENTIFIER: US 5520917 A

TITLE: Materials in the form of colored spherical fine particles

Abstract Paragraph Left (1):

A colored particulate material in the form of almost perfectly spherical fine particles comprising an organic and/or inorganic pigment coated with a hydrated metal compound over the surface thereof, the coated pigment being enclosed with an inorganic porous wall substance, process for producing the same, and a cosmetic composition comprising the same.

Brief Summary Paragraph Right (2):

The present invention relates to materials in the form of colored spherical fine particles, processes for producing the same and cosmetic compositions containing the particulate material, and more particularly to materials in the form of colored spherical line particles comprising a porous inorganic wall substance enclosing an organic and/or inorganic pigment therewith, processes for producing these particulate materials and cosmetic compositions containing the material.

Brief Summary Paragraph Right (9):

On the other hand, the beautifying effects of cosmetic compositions, especially those of makeup compositions, chiefly include a skin color correcting effect to conceal defects such as blemishes and birthmarks on the skin and a coloring effect to give attractive features. For cosmetics to exhibit these effects, heretofore used are pigments including titanium dioxide, zinc oxide and like white pigments, iron oxide red, yellow iron oxide, black iron oxide, ultramarine, Prussian blue and like inorganic coloring pigments, and tar pigments and like organic coloring pigments. These pigments are generally particulate or acicular, differ in surface properties, measure about 0.001 to about 1 micrometer in particle size and are accordingly great in the cohesive force or adhesive force of particles although satisfactory in tinting strength and hiding power, so that when incorporated into makeup compositions, they are likely to cause color irregularities and exhibit poor spreadability and an unnatural finish when the composition is applied. To overcome these problems, such pigments are used in combination with extender pigments in the form of globular or platelike particles as components of makeup compositions, or it has recently been attempted to use composite pigments prepared from such a pigment and mica, talc, silica gel, polyamide powder or like extender pigment so as to condition the composition for use, whereas satisfactory pigments still remain to be developed.

Brief Summary Paragraph Right (10):

Further to provide cosmetic compositions which exhibit an enhanced coloring effect and give an improved feel, for example, as to spreadability when applied, a colored finely particulate silica gel is proposed which is in the form of porous spherical particles containing 1 to 40 wt. % of water-insoluble coloring agent as enclosed in their voids (Examined Japanese Patent Publication No. 28605/1986), but this silica gel is not fully satisfactory with respect to the hiding power. When incorporated into skin care compositions and other cosmetic compositions, the silica gel excessively absorbs sebum from the skin to entail the problem of toughening the skin.

Brief Summary Paragraph Right (11):

Further processes are known for preparing a powder comprising capsules having a pigment enclosed therein (Examined Japanese Patent Publication Nos. 6251/1979 and 55454/1982), whereas since the pigment enclosure ratio is not sufficient, permitting the enclosed pigment to form flocs, the pigment fails to produce a brilliant color and to exhibit the desired hiding power. Moreover, the powder, which is relatively large in particle size, produces poor beautifying effects with respect to tinting

strength and hiding power and presents a rough surface over the skin when used as a component of cosmetic compositions, hence problems. The conventional processes further fail to encapsulate tar pigments and like organic pigments.

Brief Summary Paragraph Right (13):

Another object of the invention is to overcome the foregoing drawbacks of conventional cosmetic compositions of the type described, and more particularly to provide a makeup composition which produces a vivid color free from irregularities and suitable beautifying effects as in respect of tinting strength and hiding power and which is smoothly spreadable when applied and excellent in sustained beautifying effects such as retention of the color without dulling despite lapse of time.

Brief Summary Paragraph Right (15):

The other object of the invention is attained by incorporating into a cosmetic composition the above colored particulate material as it is or as treated with an oily substance or fluorine-containing resin.

Detailed Description Paragraph Right (1):

Generally, the present invention provides materials in the form of colored spherical fine particles, processes for producing such materials and cosmetic compositions containing the particulate material. The processes will be described first, followed by a description of colored particulate materials and then by a description of cosmetic compositions.

Detailed Description Paragraph Right (37):

8. When the material obtained is incorporated into cosmetic compositions, the pigment which is coated with an inorganic substance is held out of direct contact with the skin and therefore unlikely to be adsorbed by the skin or to penetrate into the skin. This obviates the likelihood of the pigment causing contact dermatitis or cutaneous disorders.

Detailed Description Paragraph Right (49):

The cosmetic composition of the present invention will be described next.

Detailed Description Paragraph Right (50):

Basically, the cosmetic composition of the invention has incorporated therein the colored particulate material, as further treated with an oily substance or fluorine-containing resin when so required.

Detailed Description Paragraph Right (51):

The colored particulate material to be incorporated into the desired cosmetic composition of the invention will be further described.

Detailed Description Paragraph Right (53):

The cosmetic composition of the invention may have incorporated therein the colored particulate material as it is or as treated with an oily substance or fluorine-containing resin. This treatment assures the desired cosmetic composition of sustained beautifying effects such as color retention without dulling despite lapse of time and effectively prevents the composition from roughening the skin. The oily substance to be used for the treatment is any of those usually used for cosmetic composition, such as liquid paraffin, squalane, Vaseline, paraffin wax, microcrystalline wax, beeswax, candelilla wax, rhodinic acid pentaerythritol ester, dimethylpolysiloxane and methylhydrogenpolysiloxane.

Detailed Description Paragraph Right (54):

Other compounds for treating the colored particulate material to be incorporated into the cosmetic composition of the invention are fluorine-containing resins such as Teflon and fluoroalkylphosphoric acid ethanolamine salts represented by the following formulae. Commercial products of such ethanolamine salts include AG530 (product of Asahi Glass Co., Ltd). ##STR1## In the above formulae, n is an integer of 6 to 18.

Detailed Description Paragraph Right (56):

The cosmetic composition of the present invention has incorporated therein the colored particulate material in an amount of 0.1 to 50 wt. % based on the whole weight of the composition. If the amount is less than 0.1 wt. %, a sufficient effect will not be obtained, whereas amounts in excess of 50 wt. % are not desirable not only because the effect then remains unchanged and also because the cosmetic composition is not usable to give a wide variety of impressions, or is lower in impact strength when in the form of a press-molded powdery product.

Detailed Description Paragraph Right (57):

The cosmetic composition of the present invention is available in various forms such as powdery, liquid, solid or gel forms. Such compositions are usable as skin care compositions including pack, sun care milk, lotion and body powder compositions, and makeup compositions including face powder, foundation, eye shadow, blush, lipstick, eye liner and eye brow compositions.

Detailed Description Paragraph Right (58):

Besides the colored particulate material, various additives which are usually used for cosmetics can be incorporated as suitably selected into the present composition insofar as they do not impair the advantages of the invention. Examples of such additives are inorganic pigments, organic pigments, gelling agents, surfactants, lower alcohols, polyhydric alcohols, purified water, preservatives, antioxidants, ultraviolet absorbers, humectants, beautifying components, perfumes, etc.

Detailed Description Paragraph Right (59):

The cosmetic composition of the invention having the colored particulate material incorporated therein produces a vivid color free from irregularities, exhibits beautifying effects such as a suitable tinting strength and hiding power and effects to block ultraviolet rays such as an antisunburn effect, is smoothly spreadable when applied and will not roughen the skin. When the present composition is a makeup composition, the composition forms a cosmetic coating which appears natural on finishing and exhibits greatly sustained beautifying effects such as color retentivity free of dulling despite lapse of time.

Detailed Description Paragraph Right (60):

The colored particulate material of the present invention is useful not only for cosmetic compositions but also for color printer toners, copying toners, coating compositions, printing inks, correction inks, leads of color pencils and aqueous or oily marking inks and as additives for resins and coloring agents for pharmaceutical products.

Detailed Description Paragraph Right (72):

Into a 100-ml Erlenmeyer flask of Teflon were placed 27 g of an NTL agent L-2102 (brand name, product of Nissan Chemical Industries, Ltd.), i.e., a mixture of an alkoxy silane and silica sol, and 10 g of isopropyl alcohol. A 4.5 g quantity of a commercial hydrophobic pigment, Color Index No. 74160 (Phthalocyanine Blue) and 35 g of anatase-type titanium dioxide (brand name, "JA-1", product of Tayca Corporation) were placed into the flask, and wetted with and dispersed in the mixture by an ultrasonic dispersing machine.

Detailed Description Paragraph Right (128):

The makeup base of the invention had a vivid color and suitable control effect, exhibited good spreadability when applied, did not roughen the skin and sustained the effects of foundation cosmetics.

Detailed Description Paragraph Right (145):

The powder of the invention achieved a high effect to block ultraviolet rays (FIG. 4), was smoothly spreadable when applied to give a uniform finish, had a natural matting effect and hiding power and further exhibited greatly sustained beautifying effects without becoming dull with time.

## CLAIMS:

6. A cosmetic composition comprised of colored almost perfectly spherical fine particles comprising an organic pigment and/or an inorganic pigment coated with a hydrated metal compound over the surface thereof, the coated pigment or pigments being enclosed with a porous layer of inorganic material, the colored almost perfectly spherical particles being prepared by a process comprising adding an organic or inorganic pigment coated with a hydrated metal compound to an aqueous solution containing at least one inorganic compound selected from the group consisting of alkali metal silicates, carbonates, and phosphates, and alkaline earth metal halides and nitrates to obtain a dispersion, and admixing with the dispersion with an aqueous solution of at least one compound selected from the group consisting of alkaline earth metal halides, inorganic acids, organic acids, inorganic acid ammonium salts, organic acid ammonium salts and alkali metal carbonates and capable of forming a water-insoluble precipitate by an aqueous solution reaction with the inorganic compound, before or after admixing the dispersion with an organic solvent

having a solubility of up to 8% in water to obtain a W/O emulsion.

7. A cosmetic composition as defined in claim 6 wherein the particles are 0.1 to 50 micrometers in size.

8. A cosmetic composition as defined in claim 6 wherein the particles are treated with at least one oily substance selected from the group consisting of liquid paraffin, squalene, Vaseline, paraffin wax, microcrystalline wax, beeswax, candelilla wax, rhodinic acid, pentaerythritol ester, dimethyl polysiloxane and methylhydrogenpolysiloxane, or at least one fluorine-containing resin selected from the group consisting of Teflon and fluoroalkylphosphoric acid ethanolamine salts.

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L9: Entry 1 of 4

File: USPT

Jun 9, 1998

DOCUMENT-IDENTIFIER: US 5762915 A

TITLE: Photochromic ultra-violet ray shield powder, method of manufacturing the same, and skin external preparation using the same

Assignee Name ( Derived ) (1):  
Shiseido Co., Ltd.Brief Summary Paragraph Right (16):According to claim 3, there is provided photochromic ultra-violet ray shield powder, which contains at least 50% of anatase type titanium oxide.Brief Summary Paragraph Right (27):Titanium oxide used as material according to the invention may be titanium dioxide lower titanium oxide, etc. These titanium oxide varieties may be used in combinations. Titanium dioxide may be of anatase type, rutile type and amorphous. Any of these varieties may be used. Suitably, at least 50% of anatase type titanium oxide may be contained because in this case satisfactory photochromic property can be obtained. The particles may be indefinite, flaky or spherical in shapeBrief Summary Paragraph Right (46):

The composition incorporating the photochromic ultra-violet ray shield powder according to the invention, may suitably incorporate, if desired, other components usually used for the compositions of cosmetics or the like. For example, it may incorporate inorganic powder, e.g., talc, kaolin, mica, sericite, white mica, gold mica, synthetic mica, red mica, black mica, rithia mica, vermicurite, magnesium carbonate calcium carbonate, diatomaceous earth, magnesium silicate, calcium silicate, aluminum silicate, barium silicate, barium sulfate, strontium, silicate, tungstate metal salts, silica, magnesium oxide, calcium oxide, zeolite, boron nitride, ceramic powder, etc., organic powder, e.g., nylon powder polyethylene powder, benzoguanamine powder, tetrafluoroethylene powder, fine crystalline cellulose, etc., inorganic white pigments, e.g., titanium dioxide, zinc oxide, etc., inorganic red pigments, e.g., iron oxide (red iron oxide), iron titanate, etc., inorganic brown pigments, e.g., gamma-iron oxide, etc., inorganic yellow pigments, e.g., yellow iron oxide, less, etc., inorganic black pigments, e.g., black iron oxide, carbon black, etc., inorganic violet pigments, e.g., mango violet cobalt violet, etc., inorganic green pigments, e.g., chromium oxide, chromium hydroxide, cobalt titanate, etc., inorganic blue pigments, e.g., ultramarine, red marine, etc., pearl pigments, e.g., mica coated with titanium dioxide, bismuth oxychloride coated with titanium dioxide, bismuth oxychloride, talc coated with titanium oxide, mica coated with colored titanium oxide, etc., metal powder pigments, e.g., aluminum powder, copper powder, etc., such organic pigments as "Red No. 201", "Red No. 202", "Red No. 204", "Red No. 205", "Red No. 220", "Red No. 226", "Red No. 228", "Red No. 405", "Orange No. 203", "Orange No. 204", "Yellow No. 205", "Yellow No. 401", "Blue No. 404", etc. and such organic pigments as "Red No. 3", "Red No. 104", "Red No. 106", "Red No. 227", "Red No. 230", "Red No. 401", "Red No. 505", "Orange No. 205", "Yellow No. 5", "Yellow No. 202", "Yellow No. 203", "Green No. 3", "Blue No. 1", zirconium, barium, aluminum lake, etc., natural dyestuffs, e.g., chlorophyll, beta-karotin, etc., various hydrocarbons e.g., squalane, liquid paraffin, vaseline, microcrystalline wax, ozokerite, serecile, cetylalcohol, hexadecylalcohol, oleilalcohol, cetyl-2-ethyl hexanoate, 2-ethylhexyl permiate, 2-octyldodecyl myristate, 2-octyldodecylgum ester neopentylglycol-2-ethyl hexanoate triglyceril iso-octanoate 2-ocylidodecyl oleate, iso-propyl myristate, triglyceril iso-stearate, coconut oil fatty acid triglyceride, olive oil, avocado oil, bees way, myristyl myristate, mink oil, ranoline, dimethylpolysiloxane, etc., oil, e.g., fat and oil, esters, higher alcohols, solders, silicone oil, etc., ultra-violet ray absorbers, anti-oxidization agents, surface active agents, moisture retainers, perfumes, water,



alcohol and viscosity increasing agents.

Detailed Description Paragraph Right (18):

Photochromic property is shown as color difference .DELTA.E in case of using rutile type titanium oxide as titanium oxide in Table 2, and in case of using anatase type titanium oxide in Table 3.

Detailed Description Paragraph Right (22):

When the anatase type is used, the photochromic property can be extremely improved compared to the case of using the rutile type. Also the color change degree is large, i.e. 660.degree. to 700.degree. C. However it is suitably about 600.degree. C. when the restoration factor is also taken into considerations.

Detailed Description Paragraph Right (25):

As is clear from Tables 4 to 6, by using anatase type titanium oxide, satisfactory color change degree can be obtained with any of the pH 8 to 10. Considering the restoration factor, however, lower calcinating temperature is rather satisfactory. The color change degree is most satisfactory with pH 8. On the other hand, the restoration factor is the better the higher pH.

Detailed Description Paragraph Right (27):

Rutile type titanium oxide has a large refractive index and said to have greater ultra-violet ray shield effect than anatase type titanium oxide provided the particle size is the same. Therefore, when the ultra-violet ray shield effect is considered, it is suitable to incorporate rutile type titanium oxide.

Detailed Description Paragraph Right (28):

Accordingly, researches were conducted on the Influence of incorporating rutile type titanium oxide in anatase type titanium oxide or the photochromic property.

Detailed Description Paragraph Right (31):

In the overall consideration of the above, it will be understood that when using the neutralization process it is suitable to use 75% or more of anatase type titanium oxide and effect a treatment about pH 8.

Detailed Description Paragraph Right (34):

Tables 10 and 11 below show the photochromic property in terms of the color difference .DELTA.E, respectively when rutile type titanium oxide is used as titanium oxide and when anatase type titanium oxide is used.

Detailed Description Paragraph Right (36):

With anatase type titanium oxide used as the material, the lower the calcinating temperature the greater the color change degree as is seen from Table 11. In addition the restoration factor is the better the lower the calcinating temperature.

Detailed Description Paragraph Right (42):

It will be understood from the above that by using the neutralization process with anatase type titanium oxide as material. sufficient photochromic property can be obtained at 560.degree. to 580.degree. C., which is lower than the temperature of 600.degree. C. thought to be necessary for imparting with the photochromic property in prior art.

Detailed Description Paragraph Right (55):

First, anatase type was investigated by varying the amount of Fe.sub.2 O.sub.3.

Detailed Description Paragraph Right (56):

It will be seen that when anatase type titanium oxide is used, excellent photochromic property can be obtained with any amount of Fe.sub.2 O.sub.3.

Detailed Description Paragraph Right (59):

As the result of the above test, it was made clear that when Using anatase type titanium oxide, it is suitable to add an active agent by 0.25 to 0.5% by weight in terms of Fe.sub.2 O.sub.3.

Detailed Description Paragraph Right (63):

From the above various experiments, it is made clear that for obtaining excellent photochromic property by calcinating at a comparatively low temperature below 700.degree. C. it is suitable to incorporate 50% or more of anatase type titanium oxide and incorporate iron, if used as active agent, by about 0.25% by weight in

terms of Fe.sub.2 O.sub.3.

Detailed Description Paragraph Right (70):

The phantom line plot II shows the relation between the specific surface area S.sub.2 and temperature T in case when anatase type titanium oxide of 100 m.sup.2 /g, obtained by the liquid phase process from Ti(O-iso-Pr).sub.4, was calcinated at various temperatures.

Detailed Description Paragraph Right (80):

Accordingly, material titanium oxide containing 50% or more of anatase type titanium oxide was actually calcinated at 700.degree. C. or below, and the ultra-violet ray shield effect of the calcinated material was investigated.

Detailed Description Paragraph Right (85):

100 g of titanium oxide (anatase type, specific surface area: 98 m.sup.2 /g) was dispersed in 2,000 ml of deionized water. Then, 10.5 g of iron chloride FeCl.sub.3 was added, and the system was stirred. Then, about 10 g of sodium hydroxide was added for neutralization to pH 8 (shield powder sample 1), pH 9 (shield powder sample 2) and pH 10 (shield powder sample 3), followed by reaction at 80.degree. to 90.degree. C. for 2 hours. Then, each system was filtered, and the residue was washed with water and dried. The dry residue was then calcinated at 600.degree. C. and disintegrated. As comparative example, calcinating was effected at 800.degree. C.

Detailed Description Paragraph Right (90):

40 g of titanium oxide (anatase type, 98 m.sup.2 /g) was dispersed in 800 ml of deionized water, then 0.41 g of iron chloride FeCl.sub.3 was added, and the system was then stirred. Then, 5 ml of 5% sodium hydroxides aqueous solution was added for neutralization. Then, 1.29 g of area was added and the system was reacted at 90.degree. to 98.degree. C. for 3 hours. Then, the samples were filtered, and the residues were washed with water and dried. Then, calcinating was done at 400.degree. C. (sample 4). 600.degree. C. (sample 5) and 700.degree. C. (sample 6), and each sample was then disintegrated. The measurement was done in the same way as with the above samples 1 to 3.

Detailed Description Paragraph Right (94):

Titanium oxide (anatase type, 100 m.sub.2 /g) was prepared such that Fe.sub.2 was 0.7%, and then calcinated at 560.degree. C. (sample 7) and 600.degree. C. (sample 8).

Detailed Description Paragraph Right (101):

Anatase type titanium oxide with a specific surface area of 100 m.sup.2 /g was produced from TiOSO.sub.4 by the liquid phase process, and photochromic ultra-violet ray shield powder was produced by using 1.5% of Fe.sub.2 O.sub.3 as an active agent.

Detailed Description Paragraph Right (106):

In a first trough 100 parts of tetra-iso-propoxy titanium was heated at about 200.degree. C., in a second trough 500 parts of water was heated at about 120.degree. C., and air was introduced from each trough as carrier gas into an electric furnace at 300.degree. C. for hydrolysis. Further, in a third trough 1.5 parts of zinc chloride was heated at about 800.degree. C., and air was introduced from the trough into the same electric furnace for surface treatment. This powder was recovered using filter cloth to obtain 40 parts of material anatase type titanium oxide with a specific surface area of 100 m.sup.2 /g.

Detailed Description Paragraph Table (3):

TABLE 3 \_\_\_\_\_ Neutralization process (anatase type, 98 m.sup.2 /g, 0.7% Fe.sub.2 O.sub.3) \_\_\_\_\_

Calcinating temperature	600	660	700	800	900	.DELTA.E after ultra-violet ray irradiation for 30 minutes (A)	.largecircle.	.largecircle.	.largecircle.	.largecircle.	.DELTA.E after leaving in 3.65	5.76	6.66
5.95	2.34	dark place for 60 minutes	Restoration factor 45%	30%	25%	15%	60%	.DELTA.E after leaving in 3.07	5.02	6.09	5.52	2.17	dark place for 120 minutes
Restoration factor 54%	39%	32%	22%	63%	.DELTA.E after leaving in 1.88	3.15	3.96	4.02	1.45	dark place for 24 hours	Restoration factor 72%	62%	56%
													43%
													75%

Detailed Description Paragraph Table (4):

TABLE 4 \_\_\_\_\_ Neutralization process pH: 8 (anatase type, specific surface area: 98 m.sup.2 /g, 0.7% Fe.sub.2 O.sub.3)

Calcinating temperature 600 660 700 800  
 .DELTA.E after ultra-violet 8.03 10.00 11.00 9.46 irradiation for 30 minutes (A)  
 .largecircle. .largecircle. .largecircle. .largecircle. .DELTA.E after leaving in  
 4.73 7.57 8.99 7.90 dark place for 60 minutes Restoration factor 42% 25% 19% 17%  
 .DELTA.E after leaving in 2.68 5.01 6.34 5.57 dark place for 24 hours (B)  
 .largecircle. X X X Restoration factor 67% 50% 43% 33%

Detailed Description Paragraph Table (5):

TABLE 5 \_\_\_\_\_ Neutralization process pH: 9 (anatase  
 type, specific surface area: 98 m.sup.2 /g, 0.7% Fe.sub.2 O.sub.3)  
 Calcinating temperature 600 660 700 800  
 .DELTA.E after ultra-violet 6.31 7.61 9.59 4.59 ray irradiation for 30 minutes (A)  
 .largecircle. .largecircle. .largecircle. .DELTA. .DELTA.E after leaving in 3.07 5.06  
 5.54 3.51 dark place for 60 minutes Restoration factor 52% 34% 48% 24% .DELTA.E after  
 leaving in 1.71 3.08 3.66 2.42 dark place for 24 hours (B) .largecircle. .DELTA.  
 .DELTA. .largecircle. Restoration factor 73% 60% 62% 48%

Detailed Description Paragraph Table (6):

TABLE 6 \_\_\_\_\_ Neutralization process pH: 10 (anatase  
 type, 98 m.sup.2 /g, 0.7% Fe.sub.2 O.sub.3)  
 Calcinating temperature 600 660 700 800 .DELTA.E after ultra-violet 6.17 7.05 8.09  
 5.28 irradiation for 30 minutes (A) .largecircle. .largecircle. .largecircle.  
 .largecircle. .DELTA.E after leaving in 2.70 4.24 5.73 4.26 dark place for 60 minutes  
 Restoration factor 57% 40% 30% 20% .DELTA.E after leaving in 1.25 2.30 3.48 2.78 dark  
 place for 24 hours (B) .largecircle. .largecircle. .DELTA. .largecircle. Restoration  
 factor 80% 68% 57% 48%

Detailed Description Paragraph Table (11):

TABLE 11 \_\_\_\_\_ Urea process (anatase type 98 m.sup.2  
 /g, 0.5% Fe.sub.2 O.sub.3) \_\_\_\_\_ Calcinating  
 temperature 600 660 700 800 900 .DELTA.E after ultra-violet 7.83 7.89 7.74 6.58 2.54  
 irradiation for 30 minutes (A) .largecircle. .largecircle. .largecircle.  
 .largecircle. X .DELTA.E after leaving in 3.26 4.19 5.18 5.54 1.16 dark place for 60  
 minutes Restoration factor 59% 47% 34% 16% 54% .DELTA.E after leaving in 2.49 3.61  
 5.01 5.23 1.04 dark place for 120 minutes Restoration factor 69% 55% 36% 21% 59%  
 .DELTA.E after leaving in 1.69 2.14 3.24 3.95 0.69 dark place for 24 hours (B)  
 .largecircle. .largecircle. .DELTA. .DELTA. .largecircle. Restoration factor 79% 73%  
 59% 40% 73%

Detailed Description Paragraph Table (12):

TABLE 12 \_\_\_\_\_ Neutralization process pH: 8 (anatase  
 type, specific surface area: 98 m.sup.2 /g, Fe.sub.2 O.sub.3 0.7%)  
 Calcinating temperature 560 580 .DELTA.E after  
 ultra-violet ray 7.16 7.38 irradiation for 30 minutes (A) .largecircle. .largecircle.  
 {E after leaving dark place 3.47 4.18 for 60 minutes Restoration factor 52% 44%  
 .DELTA.E after leaving in dark place 2.80 3.59 for 120 minutes Restoration factor 61%  
 52% .DELTA.E after leaving in dark place 1.58 2.21 for 24 hours (B) .largecircle.  
 .largecircle. Restoration factor 78% 71%

Detailed Description Paragraph Table (13):

TABLE 13 \_\_\_\_\_ Neutralization process pH: 9 (anatase  
 type, specific surface area: 98 m.sup.2 /g, Fe.sub.2 O.sub.3 : 0.7%)  
 Calcinating temperature 560 580 .DELTA.E after  
 ultra-violet ray 6.13 5.80 irradiation for 30 minutes (A) .largecircle. .largecircle.  
 .DELTA.E after leaving in dark place 2.26 2.46 for 60 minutes Restoration factor 64%  
 57% .DELTA.E after leaving in dark place 1.75 2.05 for 120 minutes Restoration factor  
 72% 65% .DELTA.E after leaving in dark place 0.93 1.18 for 24 hours (B) .largecircle.  
 .largecircle. Restoring factor 85% 80%

Detailed Description Paragraph Table (14):

TABLE 14 \_\_\_\_\_ Neutralization process pH: 10  
 (anatase type, specific surface area: 98 m.sup.2 /g, Fe.sub.2 O.sub.3 : 0.7%)  
 Calcinating temperature 560 580 .DELTA.E after  
 ultra-violet ray 6.26 7.02 irradiation for 30 minutes (A) .largecircle. .largecircle.  
 .DELTA.E after leaving in dark place 2.10 3.26 for 60 minutes Restoration factor 67%  
 54% .DELTA.E after leaving in dark place 1.35 2.75 for 120 minutes Restoration factor  
 79% 61% .DELTA.E after leaving in dark place 0.60 1.76 for 24 hours (B) .largecircle.

.largecircle. Restoration factor 90% 75% \_\_\_\_\_

Detailed Description Paragraph Table (20):

TABLE 20 \_\_\_\_\_ Urea process (anatase type, specific surface area: 100 m.sup.2 /g, 0.7% Fe.sub.2 O.sub.3)  
 \_\_\_\_\_ Calcinating temperature 400 500 .increment.E  
 after ultra-violet ray irradiation 7.14 8.94 for 30 minutes (A) .smallcircle.  
 .smallcircle. .increment.E after leaving in dark place for 3.49 3.70 60 minutes  
 Restoration factor 52% 59% .increment.E after leaving in dark place for 3.01 2.74 120  
 minutes Restoration factor 58% 70% .increment.E after leaving in dark place for 2.37  
 1.54 24 hours (B) .smallcircle. .smallcircle. Restoration factor 67% 83%

Detailed Description Paragraph Table (21):

TABLE 21 \_\_\_\_\_ Urea process (anatase type, specific surface area: 100 m.sup.2 /g, 1.5% Fe.sub.2 O.sub.3)  
 \_\_\_\_\_ Calcinating temperature 400 500 .increment.E  
 after ultra-violet ray irradiation 5.32 6.25 for 30 minutes (A) .smallcircle.  
 .smallcircle. .increment.E after leaving in dark place for 1.82 2.46 60 minutes  
 Restoration factor 66% 61% .increment.E after leaving in dark place for 1.53 2.06 120  
 minutes Restoration factor 72% 68% .increment.E after leaving in dark place for 0.83  
 1.21 24 hours (B) .smallcircle. .smallcircle. Restoration factor 85% 81%

Detailed Description Paragraph Table (22):

TABLE 22 \_\_\_\_\_ Urea process (anatase type, specific surface area: 100 m.sup.2 /g, 3.0% Fe.sub.2 O.sub.3)  
 \_\_\_\_\_ Calcinating temperature 400 500 .increment.E  
 after ultra-violet ray irradiation 6.25 6.76 for 30 minutes (A) .smallcircle.  
 .smallcircle. .increment.E after leaving in dark place for 3.43 4.10 60 minutes  
 Restoration factor 45% 39% .increment.E after leaving in dark place for 2.98 3.71 120  
 minutes Restoration factor 58% 45% .increment.E after leaving in dark place for 1.75  
 2.40 24 hours (B) .smallcircle. .smallcircle. Restoration factor 72% 64%

Detailed Description Paragraph Table (23):

TABLE 23 \_\_\_\_\_ Urea process (anatase type, specific surface area: 48 m.sup.2 /g, 0.7% Fe.sub.2 O.sub.3)  
 \_\_\_\_\_ Calcinating temperature 580 600 660 700  
 .increment.E after ultra-violet ray 8.15 7.74 6.85 9.57 irradiation for 30 minutes  
 (A) .smallcircle. .smallcircle. .smallcircle. .smallcircle. .increment.E after  
 leaving in dark 3.31 3.91 4.21 7.30 place for 60 minutes Restoration factor 59% 49%  
 39% 24% .increment.E after leaving in dark 2.44 3.31 3.79 6.80 place for 120 minutes  
 Restoration factor 70% 57% 45% 29% .increment.E after leaving in dark 1.26 1.71 1.94  
 4.23 place for 24 hours (B) .smallcircle. .smallcircle. .smallcircle. X Restoration  
 factor 85% 78% 72% 56%

Detailed Description Paragraph Table (24):

TABLE 24 \_\_\_\_\_ Urea process (anatase type, specific surface area: 100 m.sup.2 /g, 1.5% Fe.sub.2 O.sub.3)  
 \_\_\_\_\_ Calcinating temperature 560 600 660 700  
 .increment.E after ultra-violet ray 5.40 5.33 6.98 6.15 irradiation for 30 minutes  
 (A) .smallcircle. .smallcircle. .smallcircle. .smallcircle. .increment.E after  
 leaving in dark 2.16 2.39 3.22 3.62 place for 60 minutes Restoration factor 60% 55%  
 54% 41% .increment.E after leaving in dark 1.86 2.33 2.74 2.94 place for 120 minutes  
 Restoration factor 66% 56% 61% 52% .increment.E after leaving in dark 0.87 0.93 1.15  
 2.15 place for 24 hours (B) .smallcircle. .smallcircle. .smallcircle. .smallcircle.  
 Restoration factor 84% 83% 84% 65%

Detailed Description Paragraph Table (25):

TABLE 25 \_\_\_\_\_ Urea process (anatase type, specific surface area: 100 m.sup.2 /g, 3.0% Fe.sub.2 O.sub.3)  
 \_\_\_\_\_ Calcinating temperature 560 600 660 700  
 .increment.E after ultra-violet ray 4.82 5.29 5.34 5.83 irradiation for 30 minutes  
 (A) .increment. .smallcircle. .smallcircle. .smallcircle. .increment.E after leaving  
 in dark 2.77 2.69 3.49 4.44 place for 60 minutes Restoration factor 43% 49% 35% 24%  
 .increment.E after leaving in dark 2.50 2.42 3.23 4.30 place for 120 minutes  
 Restoration factor 48% 54% 40% 26% .increment.E after leaving in dark 1.16 1.48 1.99  
 2.71 place for 24 hours (B) .smallcircle. .smallcircle. .smallcircle. .smallcircle.

Restoration factor 76% 72% 63% 54% \_\_\_\_\_

Detailed Description Paragraph Table (31):

TABLE 31	_____	Material Specific	Specific surface
titanium surface	Treatment calcinating area after oxide area process	Fe.sub.2	O.sub.3
temperature calcinating	_____	Anatase	98 m.sup.2 /g
Neutral- 0.7%	700.degree. C. 39.2 m.sup.2 /g ization	Same Same	Same same 660 52.2
Same Same Same same	600 67.0 Same Same Urea same 700 40.0	Same Same	Same same 660
47.0 Same Same Same same	600 68.5	_____	

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L9: Entry 2 of 4

File: USPT

May 13, 1997

DOCUMENT-IDENTIFIER: US 5628934 A

TITLE: Photochromic color rendering regulatory composition and cosmetics

Assignee Name ( Derived ) (1):  
Shiseido Co. Ltd.

Brief Summary Paragraph Right (38):

Examples of the titanium oxide used in this invention include titanium dioxide and low-oxide of titanium, etc. These may be mixtures or of the anatase type, futile type or amorphous. And the shape of the particles of titanium oxide can be undefined shape, plate shape, and spherical shape. Average particle diameters on the order of 0.005-100 .mu.m are typical. A titanium oxide blending amount of 95.0-99.95% by weight is favorable in the case of manufacturing of titanium oxide having photochromic properties.

Brief Summary Paragraph Right (54):

In addition to the titanium oxide possessing photochromic properties mentioned above, other ingredients which are used for the composition of ordinary cosmetics, etc. can be suitably blended into the composition of this invention as necessary. Examples of such ingredients that can be blended into the composition of this invention include inorganic powders such as talc, kaolin, mica, sericite, natural mica, phlogopite, synthetic mica, lepidolite, biotite, lithia mica, vermiculite, magnesium carbonate, calcium carbonate, diatomaceous earth, magnesium silicate, calcium silicate, aluminum silicate, barium silicate, barium sulfate, strontium silicate, metal tungstenate, silica, magnesium oxide, calcium oxide, zeolite, boron nitrate, and ceramic powder, etc.; organic powders such as nylon powder, polyethylene powder, benzoguanamine powder, polytetrafluoroethylene powder and crystalline cellulose, etc.; inorganic white-type pigment such as titanium dioxide and zinc oxide, etc.; inorganic red-type pigment such as iron oxide (iron oxide red) and iron titanate, etc.; inorganic brown-type pigment such as gamma-iron oxide, etc.; inorganic yellow-type pigment such as yellow iron oxide and loess, etc.; inorganic black-type pigment such as black iron oxide and carbon black, etc.; inorganic purple-type pigment such as Mango Violet and cobalt violet, etc.; inorganic green-type pigment such as chromium oxide, chromium hydroxide and cobalt titanate, etc.; inorganic blue-type pigment such as ultramarine and prussian blue, etc.; pearl pigment such as titanium dioxide-coated mica, titanium dioxide-coated bismuth oxychloride, bismuth oxychloride, titanium oxide-coated talc, fish scale foil and blue titanium oxide-coated mica, etc.; metal powder pigment such as aluminum powder and copper powder, etc.; organic pigment such as red #201, red #202, red #204, red #205, red #220, red #226, red #228, red #405, orange #203, orange #204, yellow #205, yellow #401 and blue #404, etc.; organic pigment such as zirconium, barium or aluminum lake, red #3, red #104, red #106, red #227, red #230, red #401, red #505, orange #205, yellow #4, yellow #5, yellow #202, yellow #203, green #3 and blue #1, etc.; natural dye such as chlorophyll and .beta.-carotene, etc.; various kinds of hydrocarbons such as squalane, liquid paraffin, vaseline, microcrystalline wax, ozocerite, ceresine, cetyl alcohol, hexadecyl alcohol, oleyl alcohol, cetyl-2-ethyl hexanoate, 2-ethylhexyl palmitate, 2-octyldodecyl myristate, 2-octyldodecyl gum ester, neopentylglycol-2-ethyl hexanoate, isooctylate triglyceride, 2-octyldodecyl oleate, isopropyl myristate, isostearate triglyceride, coconut oil fatty acid triglyceride, olive oil, avocado oil, bees wax, myristyl myristate, mink oil, lanolin and dimethyl polysiloxane, etc.; fats, esters, higher alcohols, waxes, oils such as silicone oil, UV absorbers, antioxidant, preservatives, surface activate agents, humectant, perfume, water, alcohol and thickeners.

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L9: Entry 3 of 4

File: USPT

May 26, 1992

DOCUMENT-IDENTIFIER: US 5116664 A  
TITLE: Titanium-mica composite material

Assignee Name ( Derived ) (1):  
Shiseido Company Ltd.

Brief Summary Paragraph Right (4):

As is well known in the art, conventional titanated mica composite materials used, as a pigment, in, for example, cosmetics, paints, and plastics, comprise mica and a titanium dioxide layer formed coated on the surface of the mica, because these conventional titanated mica composite materials have a pearl gloss and various interference colors. These titanated mica pigments are generally prepared as disclosed in Japanese Examined Patent Publication (Kokoku) No. 43-25644 by hydrolyzing an aqueous solution of the inorganic acid salt of titanium (e.g., titanyl sulfate) in the presence of mica to deposit hydrous titanium oxide on the surface of mica, followed by heating, although these titanated mica pigments also can be prepared by a vacuum deposition method. As the starting mica, muscovite is generally used, but biotite or other mica can be also used. The mica is previously subjected to aqueous grinding and sifting to obtain powder particles having a uniform particle size. The resultant titanated mica pigments exhibit various interference colors, depending upon the thickness of the titanium dioxide layer coated on the surface of the mica. The interference colors are generally silver when the titanium dioxide content is 10% to 26% by weight, gold when the titanium dioxide content is 26% to 40% by weight, red, blue, and green when the titanium dioxide content is increased from 40% by weight to 50% by weight, and higher order interference colors when the titanium dioxide content is 50% to 60% by weight.

Brief Summary Paragraph Right (6):

Various kinds of color pigments such as iron oxides, ferric ferrocyanide, chromium oxides, carbon black, and carmine have been heretofore incorporated into the above-mentioned titanated pigments to obtain various colored appearance. However, the stabilities of, for example, the safety, light resistance, chemical resistance, solvent resistance, and heat resistance of these colored titanated mica pigments largely depend upon the properties of the color pigments incorporated therein. For example, blue titanated mica pigments mainly containing ferric ferrocyanide result in undesirable color fading in an alkaline solution and are degraded and faded at a temperature of 200.degree. C. to 300.degree. C. In addition, although green titanated mica pigments comprise a mixture of iron oxide and ferric ferrocyanide or chromium oxides, these pigments have a poor, for example, chemical resistance and heat stability, and have possible disadvantages from the safety point of view due to the toxicity of the chromium. Thus, the application ranges of these pigments are limited. Furthermore, blue or green organic pigments such as phthalocyanine blue, brilliant blue FCF aluminum lake, Quinizarin green, and phthalocyanine green have a poor safety and, for example, heat resistance, light resistance, and chemical resistance, and therefore, these organic pigments are not substantially used for coloring pearl gloss materials. On the other hand, red titanated mica composite pigments containing carmine are color faded due to light. Furthermore, the colored titanated mica pigments containing the color pigments cause color segregation or foreign odor generation in a solvent or various compositions (e.g., cosmetics) when color pigments are incorporated.

Detailed Description Paragraph Right (19):

The X-ray diffraction pattern (Cu-K.alpha. line) of the resultant blue pearl gloss material was as shown in FIG. 2. As is clearly observed in FIG. 2, a peak at a diffraction angle (i.e., Bragg's angle 2.theta.) of approximately 25.3.degree. is present in addition to the diffraction peaks of the mica. This peak corresponds to

(101) of the strongest peak of the anatase type titanium dioxide.

Detailed Description Paragraph Right (26):

A 50 g amount of the resultant titanated mica was dispersed in 500 ml of an 8.times.10.sup.-3 M aqueous solution of stannous chloride and, after filtration, was again dispersed in 500 ml of a 5.times.10.sup.-4 M aqueous solution of palladium chloride. After filtration, 50 g of the activated titanated mica obtained was dispersed at a temperature of 80.degree. C. and a pH of 8-10 for one hour in 1200 ml of an electroless nickel plating bath comprising 10 g/l sodium hypophosphate, 100 g/l sodium citrate, 50 g/l ammonium chloride, and 30 g/l nickel chloride. Thereafter, the mica was dispersed for a further one hour after adding 1200 ml of 35% formalin. After filtration and washing with water, the resultant product was dried at a temperature of 150.degree. C. to obtain 54 g of a green pearl gloss material having both a brilliant appearance color and interference color.

Detailed Description Paragraph Right (27):

The amounts of the titanium dioxide and the metallic nickel coated over the mica in the green pearl gloss material obtained above were determined in the similar manner as in Example 1.

Detailed Description Paragraph Right (28):

As a result, it was found that the green pearl gloss material comprised 98.6 parts of titanium dioxide and 16.2 parts of metallic nickel over 100 parts of the mica.

Detailed Description Paragraph Table (1):

TABLE 1 \_\_\_\_\_ Example No. Color tone HV/C\*  
 \_\_\_\_\_ 1 Blue 9.02 PB 5.68/4.61 2 Green 2.30 G  
 4.13/3.29 3 Blue 4.23 B 5.18/3.29 4 Reddish purple 6.13 RP 4.31/3.16 5 Blue 5.34 PB  
 3.41/10.66 6 Red 5.25 RP 3.36/6.75 7 Gold 4.98 Y 7.33/10.41 8 Silver 7.50 Y 8.62/2.13  
 9 Blue 7.24 PB 7.38/8.15 \_\_\_\_\_ \*The color tone was  
 determined in terms of hue (H), brightness (V), and chroma in a powdercell method by  
 a color analyzer 607 (manufactured by HITACHI, LTD.).

Detailed Description Paragraph Table (2):

TABLE 2 \_\_\_\_\_ Comparative Commercially Present  
 Pigment Available Pigment \_\_\_\_\_ Products of Examples  
 4 and 6 Cloisonne Red Products of Examples 1, 3, Cloisonne Blue 5, and 9 Product of  
 Example 2 Cloisonne Green Product of Example 7 Cloisonne Gold

Detailed Description Paragraph Table (3):

\_\_\_\_\_ Ferric Iron ferro- Composition (%) TiO.sub.2  
 Mica oxide cyanide Carmine \_\_\_\_\_ Cloisonne Red 36-42  
 56-62 -- -- 1.5-3 Cloisonne Blue 44-49 48-54 -- 2-5 -- Cloisonne Green 44-48 44-48  
 4-7 1-3\* -- Cloisonne Gold 32-38 58-64 2-6 -- --  
 \_\_\_\_\_ \*1 Chromium oxide

Detailed Description Paragraph Table (4):

TABLE 4 \_\_\_\_\_ Example No. or Commercially Available  
 Light Heat Stability Pigment Stability 200.degree. C. 300.degree. C. 400.degree. C.  
 \_\_\_\_\_ Example 1 0.2 0.13 0.26 3.38 Example 2 0.3 0.1  
 0.31 4.41 Example 3 0.3 0.1 0.22 3.35 Example 4 0.2 0.1 0.21 4.33 Example 5 0.2 0.1  
 0.28 5.00 Example 6 0.2 0.11 0.19 4.62 Example 7 0.1 0.1 0.36 4.13 Example 8 0.2 0.16  
 0.23 4.61 Example 9 0.2 0.1 0.35 4.77 Cloisonne Red 35.3 3.5 26.2 45.0 Cloisonne Blue  
 5.2 3.2 36.4 37.2 Cloisonne Green 6.0 0.2 0.6 7.8 Cloisonne Gold 0.4 0.3 0.6 1.2

Detailed Description Paragraph Table (5):

TABLE 5 \_\_\_\_\_ Example No. or Commercially Alkali  
 Available Dispersion Stability\*.sup.1 Sta- Pigment 5 min 10 min 30 min 60 min  
 bility\*.sup.2 \_\_\_\_\_ Example 1 + + + + ++ Example 2 +  
 + + + ++ Example 3 + + + + ++ Example 4 + + + + ++ Example 5 + + + + ++ Example 6 + +  
 + + ++ Example 7 + + + + ++ Example 8 + + + + ++ Example 9 + + + + ++ Cloisonne Red  
 .+- .+- .- - - Cloisonne Blue .+- .+- .- - - Cloisonne Green + + .+- .- .+-  
 Cloisonne Gold + + + + ++ \_\_\_\_\_ \*.sup.1 Evaluation  
 Criteria +: No sedimentation and good dispersibility .+-.: Sedimentation progressing  
 with color segregation -: Complete sedimentation with color segregation \*.sup.2  
 Evaluation Criteria ++: No color tone change and extremely stable .+-.: Gradually  
 fading and becoming whitish -: Faded and became white